High Resolution Study of the Thermomechanical Behavior of Al(0.5wt% Cu) Thin Films by Scanning X-Ray Microdiffraction (μSXRD)

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INTRODUCTION

Materials properties such as strength, resistance to fatigue, and failure ultimately depend on the microstructural features of the material, such as grains, grain boundaries, inclusions, voids and other defects. The so-called mesoscopic length scale (approximately between 0.1 and 10 μm) is receiving increasing attention, both theoretically and experimentally, in order to understand the mechanical behavior of polycrystalline samples as they experienced various constraints. Compared to single crystals, polycrystals are a highly inhomogeneous medium where local stress and structure variations are likely to play an important role in the overall macroscopic behavior of the material. In the present study, we are applying the Scanning X-Ray Microdiffraction (μSXRD) technique developed at the ALS [1,2] to probe local strain/stress and grain orientation in Al(0.5wt% Cu) thin films and compare the results with those obtained with conventional averaging techniques such as wafer curvature.

EXPERIMENTAL

The samples investigated are sputtered Al (0.5 wt.% Cu) thin film test structures originally designed for electromigration studies. The patterned lines, passivated with 0.7 μm of SiO_2 (PETEOS), have dimensions 0.7 or 4.1 μm in width, 30 μm in length and 0.75 μm in thickness. Ti shunt layers are present at the bottom and the top of the lines. A 100 x 100 μm bond pad on the chip with a thin Ti underlayer is used to simulate a bare blanket film.

The samples were scanned under a submicron size white X-ray beam and at each step a white beam Laue diffraction pattern in reflective geometry was collected. The patterns were analyzed in order to yield the orientation and deviatoric strain/stress tensor under each illuminated points of the samples. The outputs of the analysis are grain orientation and strain/stress maps.

The bond pad (blanket film) was thermally cycled between 25°C and 345°C in 40° steps. At each temperature increment, a 15x15 μm area of the film was scanned with the focused white x-ray beam in 1 μm steps. A 0.7 μm wide line and 4.1 μm wide line were scanned in 0.5 μm intervals at room temperature. In addition, a 0.7 μm line was mapped in 0.5 μm steps across the line and 1 μm steps along the line at several temperatures during a cycle between 25°C and 305°C.

RESULTS

A 5x5 μm area preliminary scan of the blanket film shows that while the film is (111) textured within 3°, the deviatoric stresses $\sigma_{xx}' \neq \sigma_{yy}'$ (In the x,y,z orthogonal coordinate system, z is the out-of-plane direction). In particular, at the granular and subgranular level, the stress can depart significantly from biaxiality. However, if we average the data over the 5 × 5 µm scanned area, biaxiality is retrieved, i.e.: $<\sigma'_{xx}> \approx <\sigma'_{yy}>$. The average biaxial stress of the film can be computed assuming that the out-of-plane average total stress $<\sigma_{zz}> = 0$. Expressing the total average stress matrix as the sum of the deviatoric and hydrostatic stress components one can show that the average biaxial stress $<\sigma_b> = <\sigma_{xx}> = <\sigma_{yy}> = <\{(\sigma'_{xx}+\sigma'_{yy})/2\} - \sigma'_{zz}>$.

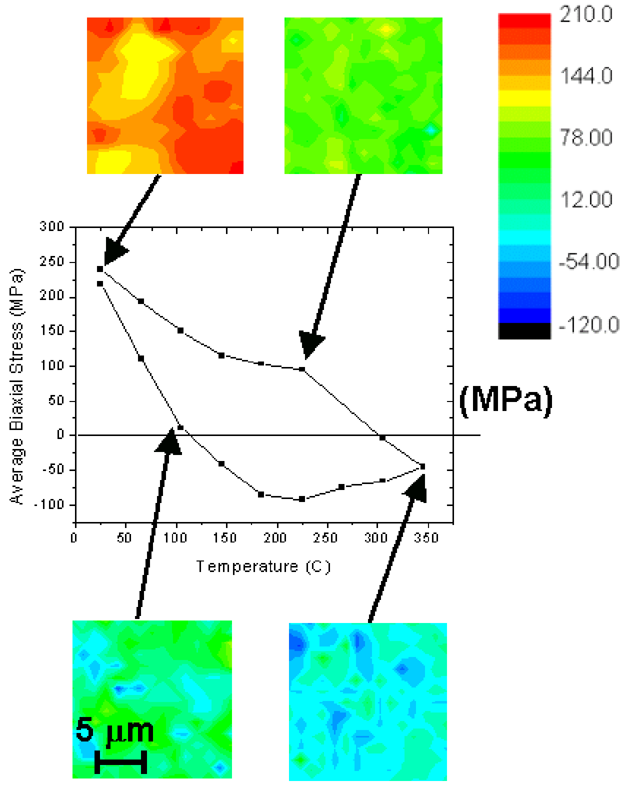

Figure 1. Thermal cycling results on a 15×15 µm area of an Al(Cu) bond pad (blanket film) showing the averaged biaxial stress component $<\sigma_b>$ versus temperature. The insets show detailed stress distribution in the film at different temperatures (The 2D maps are a plot of $-\sigma'_{zz} = \sigma'_{xx} + \sigma'_{yy}$ as a measure of the in-plane stress). Note the blue regions of compressive stress in the 105 ºC map, while on average the stress is still in the tensile regime.

A plot of the average biaxial stress in the blanket film obtained by x-ray microdiffraction during a thermal cycle between 25°C and 345°C is shown in Fig. 1. The stresses in approximately 130 grains in a 15µm x 15µm region were averaged to give the results shown in the figure. Though our temperature range is about 100°C smaller, the temperature cycling curve using microdiffraction is very similar to that reported by Venkatraman et al [3] using wafer curvature measurements on an Al(Cu) films with thickness 1 µm. The film is in tension at room temperature, with an average biaxial stress of 230 MPa. The measured stress is caused by mismatch between the thermal expansion coefficients of the aluminum and the silicon substrate. Upon heating, the higher thermal expansion coefficient of the aluminum film relaxes the tensile stress before driving the film into compression. From Fig. 1 the experimentally determined initial thermoelastic slope is $(d\sigma / dT) = 2.53$ MPa / °C. The theoretical thermoelastic slope was calculated to be 2.34 MPa / °C, in reasonable agreement with the experimental value.

The real advantage of using µSXRD is however the possibility to obtain orientation and strain/stress information at a very local level, allowing for the study of mechanical properties of thin films with new visibilities. For instance, it provides a simple and straightforward explanation to the early departure from linearity of the thermoelastic slope during heating (seen in [3] as well as in the present data), even before the film enters in the compressive state. At room temperature, the stress is highly inhomogeneous with regions sustaining more tensile stress than others. Upon heating some regions of the film become compressive while the average biaxial stress is still in the tensile regime. Even though the average stress is zero at about 100^{o}C some grains have already reached their yield stress and deformed and the heating curve departs from linearity. The temperature at which the curve departs from linearity is quite variable for different films and depends on detailed process parameters.

Similar local variations of the stress are observed in the passivated lines. These local variations increase with decreasing line width while the area of the hysterisis of the stress-temperature curves decreased. The hysteresis area is typically a measure of the amount of plastic deformation experienced by the material. In the case of submicron sized lines, the passivation layers introduce additional constraints to the dislocation motions, as compared to the case of unpassivated blanket films [4]. These local stress variations which do not always follow grain boundaries but can be intragranular, are quite surprising in an elastically isotropic material as aluminum. However these differences can be explained by differences in grain sizes and grain-to-grain interactions [5].

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REFERENCES

1. A. A. MacDowell, R. S. Celestre, N. Tamura, R. Spolenak, B. Valek, W. L. Brown, J. C. Bravman, H. A. Padmore, B. W. Batterman, and J. R. Patel, Nucl. Instr. & Meth. In Phys. Res. **A 467-468**, 936 (2001).
2. N. Tamura, R. Spolenak, B.C. Valek, A. Manceau, N. Meier Chang, R.S. Celestre, A.A. MacDowell, H.A. Padmore and J.R. Patel, Rev. of Sci. Instr., **73** (2002) in press.
3. R. Venkatraman, J. C. Bravman, W. D. Nix, P. W. Davies, P. A. Flinn, D. B. Fraser. J. of Electron. Matls. **19**, 1231 (1990).
4. B.C. Valek, N. Tamura, R. Spolenak, A.A. MacDowell, R.S. Celestre, H.A. Padmore, J.C. Bravman, W.L. Brown, B. W. Batterman and J. R. Patel, Mat. Res. Soc. Symp. Proc., **673**, P.7.7.1 (2001).
5. R. Spolenak et al. (2002) this compendium.

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